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THE EFFECT OF NUCLEATION OF SURFACE SLIP ON THE
FLOW AND FRACTURE OF BERYLLIUM

by

K. Snowden and J. Zeiger

January 22, 1963 to April 21, 1963

THE FRANKLIN INSTITUTE
LABORATORIES FOR RESEARCH AND DEVELOPMENT

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I.   INTRODUCTION

The immediate objective of the current research program is to make comparative microstrain and tensile experiments on the flow and fracture of polycrystalline Be specimens after various thermal and mechanical treatments. This report presents the preliminary results on the influence of annealing, sprinkling the surface with carborundum powder, Cu plating and strain cycling on the flow and fracture of polycrystalline hot-pressed Be.

II.   SPECIMEN PREPARATION

Specimens were prepared from rods of hot-pressed Be of specification QMV-N50 and size $\frac{1}{4}$" dia. by 6" long obtained from the Brush Beryllium Co. The specimens were shaped and cut to size in a spark-discharge apparatus which was designed and built at the Institute. The rods were machined into standard tensile shapes by the use of an appropriately shaped wheel in the spark machine. The rods, which were found to be 0.010" oversize, were first cut to 0.140" dia. at the rate of 0.001" of radius per minute using the coarse spark discharge. The final gauge diameter was 0.113" and was produced by three equal cuts of 0.009" using succeedingly finer capacitances in the discharge circuit. The final size of the specimens was 3/4" shoulder length and 11/16" gauge length. The samples were then polished using the glycerol solution from the paper "Electrolytic and Chemical Polishing", by Jacquet, Metallurgical Reviews, 1, 2 (1956). The surface of the beryllium after polishing 0.002" off the diameter showed no visible sign of twins or cracks when viewed at x 500 with an optical microscope.

Specimens were tested in the following conditions: PT1 as received, PT2 as received and strain cycled; PT3 vacuum annealed at 750°C; PT5 vacuum annealed at 850°C and sprinkled with carborundum powder, average particle size 483 μ, from a height of 1"; PT6 argon annealed at 850°C; PT7 argon annealed, electrochemically plated with copper and finally annealed at 850°C in an argon atmosphere.

III. EXPERIMENTAL PROCEDURE

Comparative microstrain and tensile experiments were carried out on Be specimens which were gripped in a straining jig mounted in an Instron machine as shown in Figure 1. Differential transformers were used to measure the displacements across a proving ring (for stress measurement) and the gauge length of the specimen (for strain measurement). The changes in output signals of the transformers corresponding to displacements across the proving ring and the gauge length were amplified and fed to a Mosley X-Y recorder which produced a continuous record of the stress-strain behavior to fracture. A number of different strain ranges were available so that the strain sensitivity could be increased to study anelastic effects at different stages of the experiment. The results of these measurements are given in Figures 2-5.

IV. EXPERIMENTAL RESULTS

(i) Microstrain Measurements

The effects of the various pre-treatments on the microstrain region of the stress-plastic strain curves for Be are shown in Figure 2. The elastic portion of the strain has been subtracted from the total strain in each case. The curves show that an anneal in vacuum at 750°C drastically reduced the micro-yield stress from 8 Kg/mm^2 to 1.7 Kg/mm^2 and generally lowered the flow stress. Sprinkling the surface of an annealed specimen with carborundum powder (mean diameter 483μ) raised the flow stress above that of the as-annealed specimen. The copper plated specimen behaved similarly to the as-annealed specimen. Cyclic straining prior to tensile testing significantly increased the strain hardening of the as-received specimen in contrast to the effects produced by the other treatments.
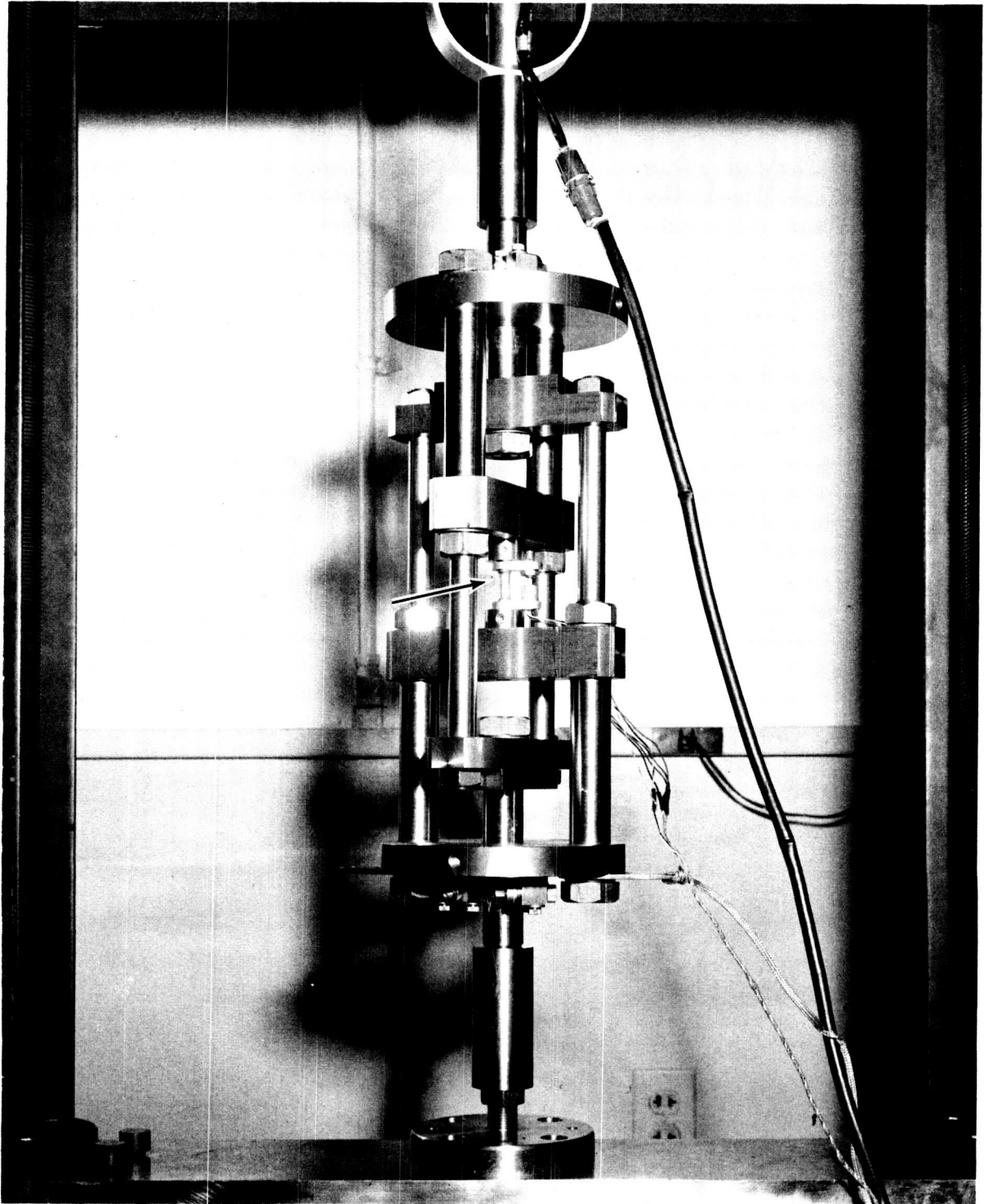
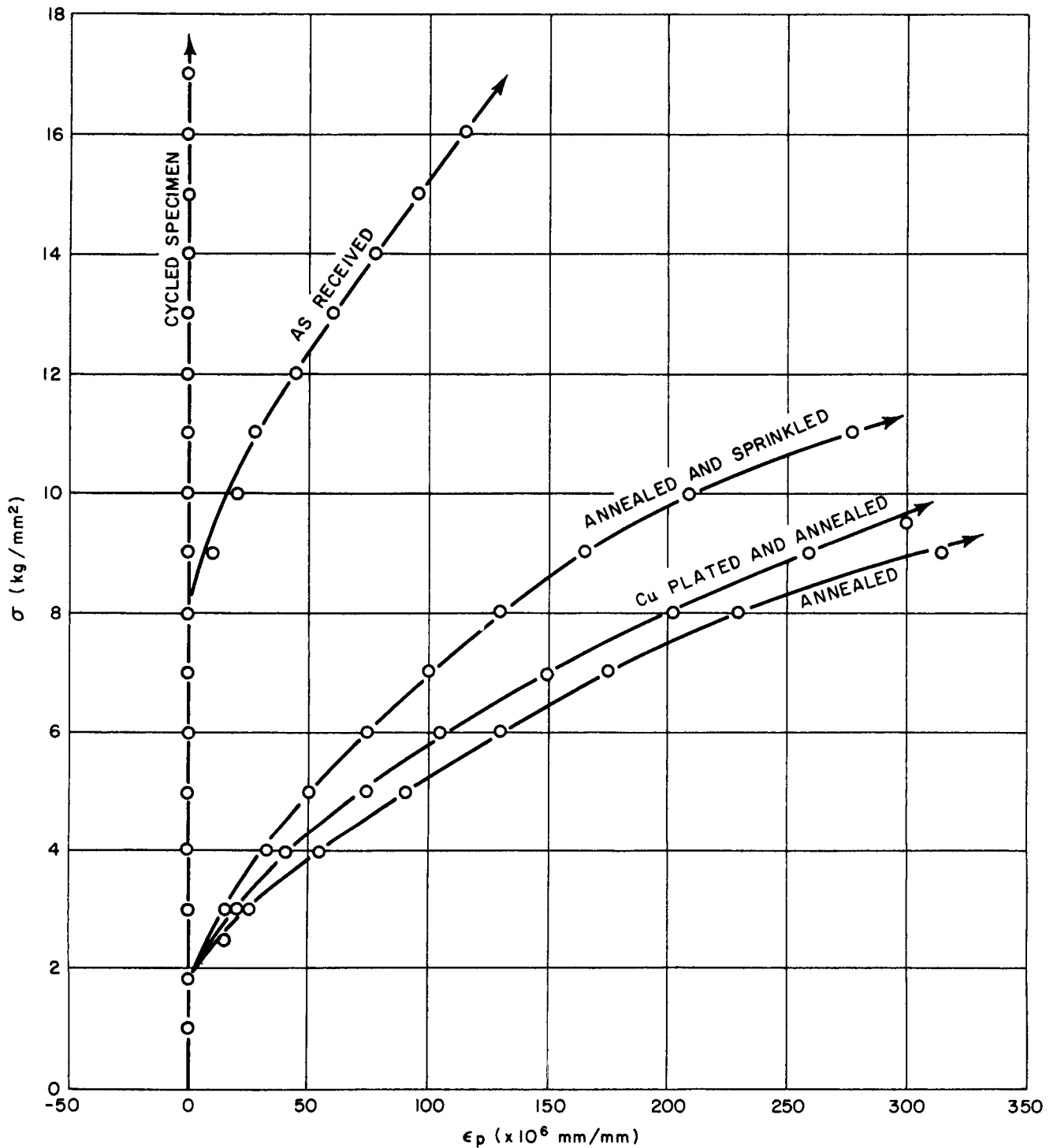*Fig. 1 - STRAINING JIG WITH SPECIMEN POSITION SHOWN.*

Fig. 2 - STRESS-PLASTIC STRAIN CURVES IN MICROSTRAIN REGION FOR HOT PRESSED POLYCRYSTALLINE Be IN THE CONDITIONS INDICATED.
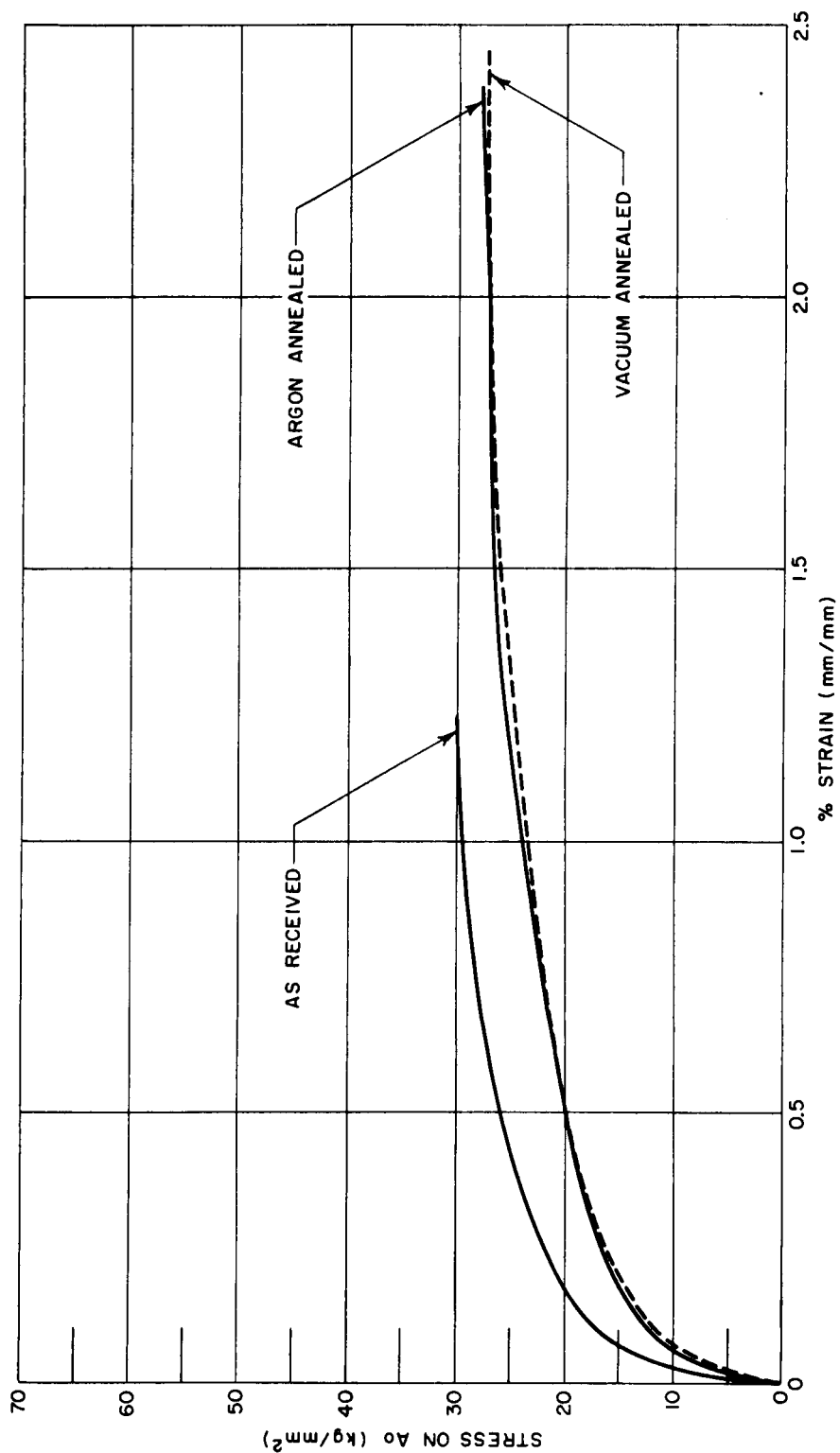
Fig. 3 - *THE STRESS-STRAIN CURVES TO FRACTURE FOR Be SPECIMENS IN THE AS-RECEIVED, ARGON ANNEALED AND VACUUM ANNEALED CONDITIONS.*
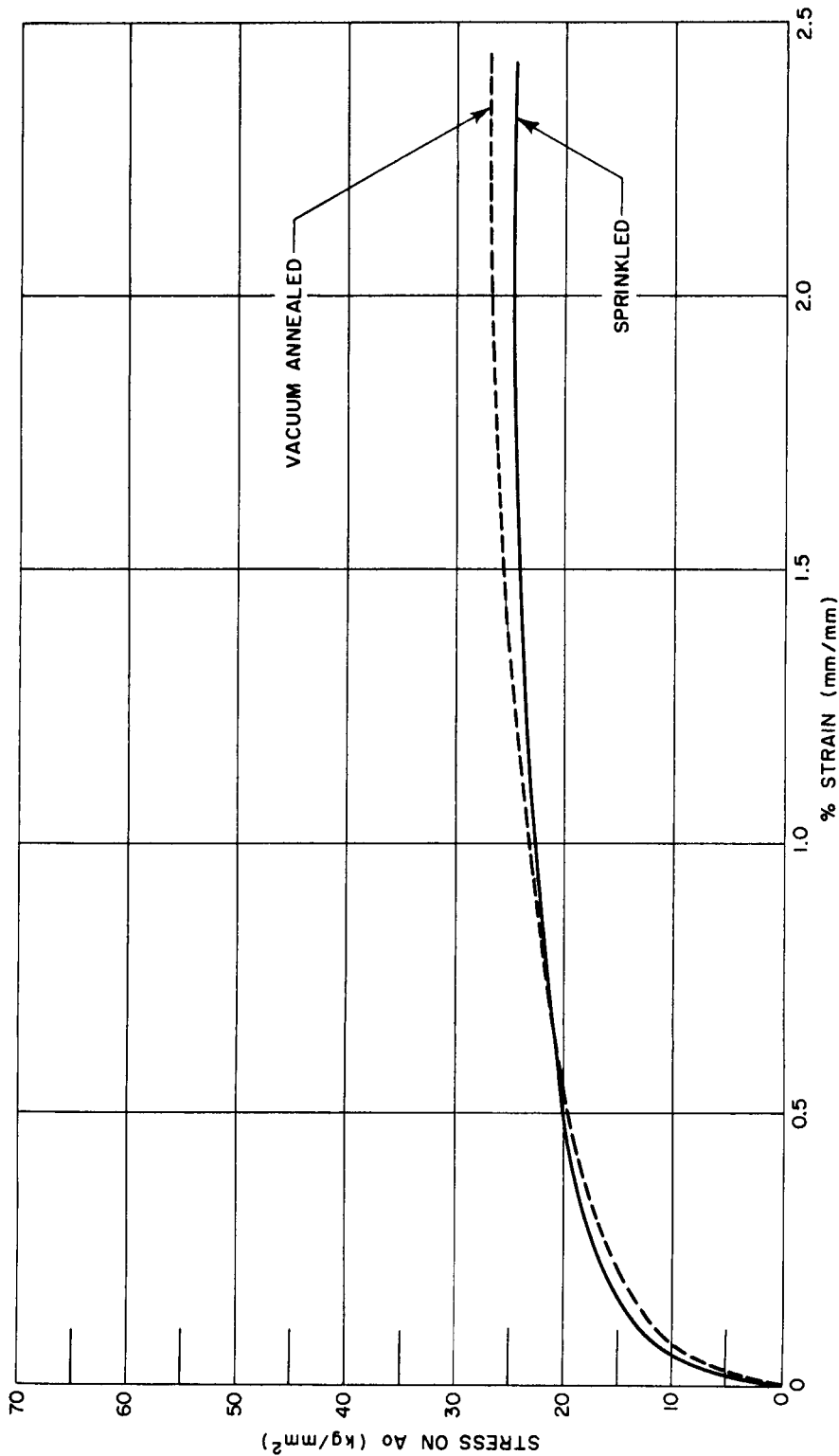
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Fig. 4 - THE STRESS-STRAIN CURVES TO FRACTURE FOR Be SPECIMENS IN THE SPRINKLED AND IN THE VACUUM ANNEALED CONDITIONS.
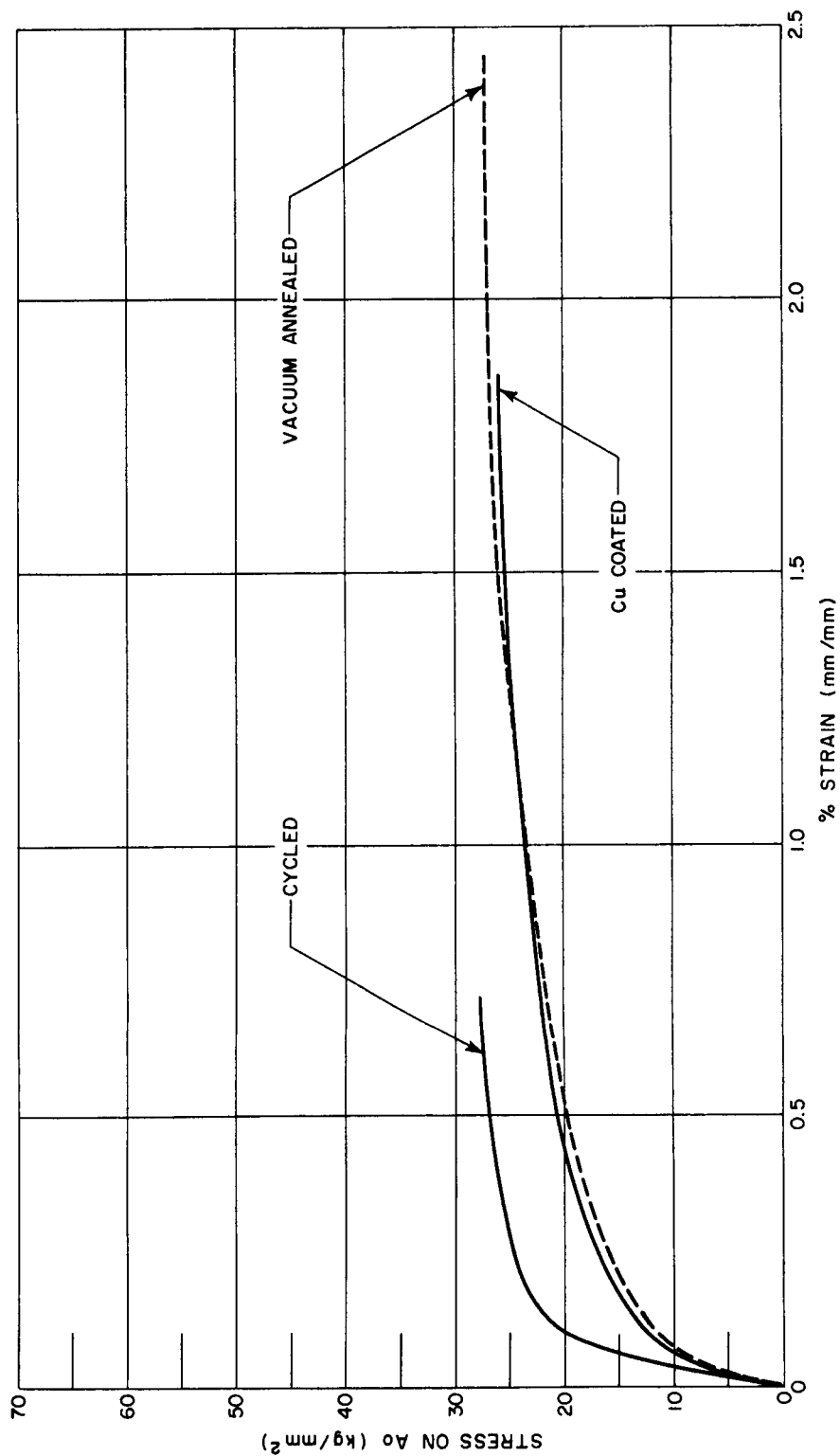
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Fig. 5 - THE STRESS-STRAIN CURVES TO FRACTURE FOR Be SPECIMENS IN THE CYCLED, IN COATED AND VACUUM ANNEALED CONDITIONS.

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Table 1

THE INFLUENCE OF SPECIMEN CONDITION ON THE MICRO-YIELD STRESS AND THE
PLASTIC STRAIN AT 9 Kg/mm^2

Specimen Condition	MYS	$(x\ 10^{-6}\ in/in)$ ϵ_p at 9 Kg/mm^2
Annealed	1.7 Kg/mm^2	315
Annealed and Cu Plated	1.7 Kg/mm^2	260
Annealed and Sprinkled	1.7 Kg/mm^2	165
As-received Condition	8.0 Kg/mm^2	15
As-received and Strain Cycled	> 9 Kg/mm^2	0

The micro-yield stress (M.Y.S.), and the plastic strain, ϵ_p, at 9 Kg/mm^2 are given in Table 1 for the various treatments.

The apparent Young's Modulus for Be was determined from the elastic portion of the stress-strain curves near zero stress. The value obtained was 30,000 Kg/mm^2 which is in good agreement with the values given by standard methods[1].

(ii) Frictional Force Measurements

The stress, σ_e, required to overcome the frictional force opposing the motion of dislocations was estimated by two methods: (a) by the length of the elastic loading and unload curves[2], and (b) by the slope of the curve of the irreversible work done in forming an anelastic loop vrs. the non-elastic strain at zero stress[2-5]. Although the detailed analysis is not yet complete, an estimate using method (a) indicates that σ_e is the approximately 1.5 to 2.0 Kg/mm^2 for the annealed, sprinkled, and Cu plated conditions. These figures may be compared with the corresponding stress of 0.035 Kg/mm^2 for Al single crystals[2], 0.10 Kg/mm^2 for Cu single crystals[3] and 2.1 Kg/mm^2 for polycrystalline zone refined iron[4].

(iii) The Stress-Strain Curves

The features of the macroscopic stress-strain curves to fracture
are shown in Figures 3-5 for the various treatments. The supplier[1] states
that the minimum guaranteed elongation for the basis material is one
percent. Figure 3 shows that annealing the as-received specimen either
in argon or in vacuum doubled the ductility with only a small reduction in
the stress at fracture. The stress-strain curve for the specimen annealed
in argon closely follows that for the specimen annealed in vacuum.

The effect of surface damage produced by sprinkling the surface
of an annealed specimen with carborundum powder is shown in Figure 4. As
mentioned in connection with the microstrain observations, the initial
flow stress of the sprinkled specimen lay above that for the annealed
specimen; however, after about $\frac{1}{2}\%$ strain the curves crossed over and
both specimens failed at approximately the same strain.

In Figure 5, the stress-strain curves for the specimen in the
cyclic strain hardened condition and the specimen in the Cu plated and
annealed condition are compared with the stress-strain curve for the specimen
in the as-annealed condition. The behavior of the Cu plated specimen
closely followed that of the as-annealed specimen except that fracture
intervened after less strain. The specimen subjected to cyclic straining
prior to tensile testing exhibited a high initial flow stress and fracture
occurred at a third of the strain at fracture of the as-annealed specimen.

The stress and strain at fracture are summarized in Table 2.

Table 2

THE EFFECT OF SPECIMEN CONDITION ON THE STRESS AND STRAIN AT FRACTURE

Test	Specimen Condition	Stress at Fracture	Strain at Fracture
(1)	As-received	29.5 Kg/mm^2	1.25%
(2)	Vacuum annealed	27.0 Kg/mm^2	2.45%
(3)	Argon annealed	27.5 Kg/mm^2	2.40%
(4)	Vac. annealed and sprinkled	25.0 Kg/mm^2	2.45%
(5)	As-Received and cycled	26.5 Kg/mm^2	0.70%
(6)	Argon annealed, Cu plated and annealed	26.0 Kg/mm^2	1.85%

Examination of the fractured specimens with the optical microscope revealed the presence of a large number of cracks, such as shown in Figures 6 and 7, for the as-received and the strain cycled specimens, respectively.

V. DISCUSSION

The interpretation of the present experiments on the influence of thermal and mechanical treatments on the flow and fracture of hot-pressed Be must be of a tentative nature since only one specimen within each condition was tested. However, the close similarity of the stress-strain curves of the specimens annealed in vacuum and in argon, Figure 3, gives some assurance that the variation in behavior from one specimen to another in the same nominal condition should not be too great.

Annealing and cyclic straining the as-received material produced the largest effects on the flow and fracture behavior. In the former treatment, the beneficial effect of annealing suggests that the existing dislocation configurations in the as-received material are responsible for the limited ductility.
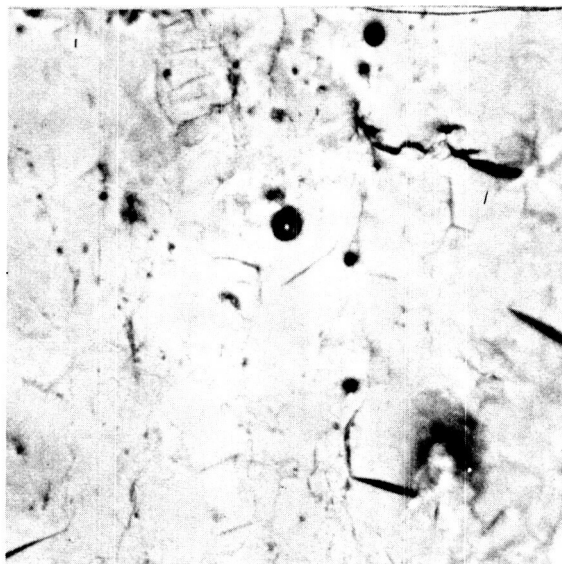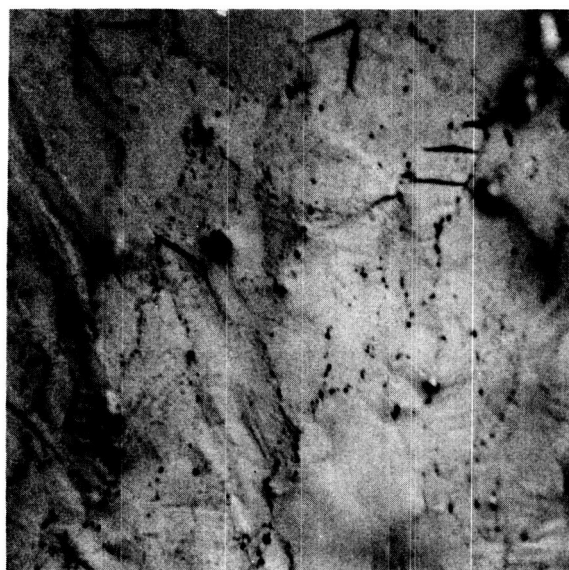
Fig. 6 - SPECIMEN PT1 AFTER CYCLING. Mag. 500X.

Fig. 7 - SPECIMEN PT2 AFTER TENSILE TEST. Mag. 250X.

The effect of prior strain cycling is in contrast to the effect
produced by strain cycling Zn single crystals which can be hardened
using this method to stresses appreciably greater than that associated
with tensile fracture[6]. In the case of the Be specimens, the cyclic
straining may have introduced small cracks or twins which would reduce
the ductility in the subsequent tensile test.

The production of surface damage initially increased the flow
stress above that of the as-annealed specimen, but after about 0.5%
strain, the flow stress fell slightly below that of the annealed specimen.
This latter behavior is in accord with recent observations on the effect
of sprinkling on MgO crystals[7].

The presence of the Cu plate had little influence on the stress-
strain curve but did reduce the ductility by 25% compared to that of
the annealed uncoated specimen. This reduced ductility is in line with
the observation of Gilman[8] that an electrodeposited layer of Cu on Zn
single crystals made them more brittle at room temperature.

VI. SUMMARY

The limited number of experiments carried out so far suggests
the following tentative conclusions:

(a) Annealing the as-received hot-pressed Be rod at 750-850°C
either in vacuum or in argon doubles the ductility, reduces the micro-
yield stress from 8 to 1.7 Kg/mm^2, and lowers the flow stress.

(b) The frictional force opposing the movement of dislocations
in the annealed, sprinkled or Cu plated specimens was estimated to be
1.5 to 2.0 Kg/mm^2.

(c) Surface damage introduced by sprinkling an annealed specimen
with carborundum powder produced only slight changes in the stress-strain
curve compared with that of the annealed specimen.

(d) The presence of a Cu electrodeposited layer reduced the ductility by 25% compared to the uncoated annealed specimen.

(e) Prior cyclic straining of an as-received specimen limited the ductility in a subsequent tensile test to one half of that of the as-received specimen and to one third of that of the as-annealed specimen.

(f) The apparent value of Young's modulus of polycrystalline Be was measured in the microstrain region near zero stress and found to be 30,000 Kg/mm^2, which is in good agreement with values published in the literature.

J. Zeiger
Research Physicist

K. U. Snowden
Senior Research Metallurgist

H.F.G.Wilsdorf
Technical Director

Approved by:

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